

Application of a High-Resolution X-Ray Fluorescence Analyzer

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Introduction

We have developed a high-resolution x-ray fluorescence analyzer based on the principle of active optics. It combines a resolution of ca. 5 eV with a tunability over several keV and a wide solid-angle coverage (ca. 2 by 5 degrees). To date, this analyzer has been used in near-edge spectroscopy of gallium in GaAs, and for the detection and chemical speciation of trace amounts platinum in soot from an diesel engine. The latter application illustrates the use of the analyzer to enhance the signal-to-background ratio in trace-element x-ray fluorescence analysis.

Methods and Materials

The analyzer is shown schematically in Fig. 1. In it, a strip of silicon is bent by an axial force from two pushers at its ends, and eight correctors act from above to bring the shape of the bent crystal to approximate a logarithmic spiral.

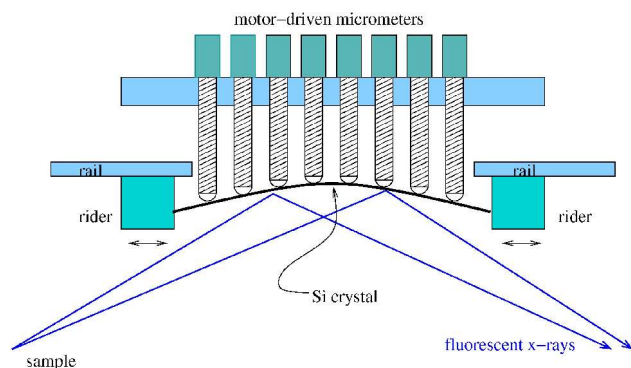


Fig 1: Schematic overview of the active-optic analyzer. Two pushers provide the main bending, and a row of correctors serve to approximate a logarithmic spiral

A more detailed description of the device, its underlying theory, and adjustment procedures may be found elsewhere [1,2].

The sample consisted of soot collected from the exhaust of a diesel engine burning a fuel with a platinum-based additive that was tested for the purpose of facilitating clean combustion.

The concentration of platinum in the soot was about 100 ppm, and the chemical speciation (oxidation state, dispersed or in the form of nanoparticles, etc.) was unknown. A small speck of this soot containing 10^{12} to 10^{13} atoms was placed into the x-ray beam of the 11-ID-D station of the APS. The incident photon energy was scanned over the Pt L₃ edge, and the Pt L_{α1} fluores-

cence was detected using two silicon drift detectors (Vortex), one directly and one with the analyzer. The purpose of the analyzer in this application was to enhance the energy resolution by a factor of about 50 (250 eV for the drift detector, 5 eV for the analyzer), and thus reduce the background of elastically or Compton-scattered photons, while keeping the fluorescent line.

Results

Whereas the detector with the analyzer recorded a clear signature of platinum in the form of an absorption edge (see Fig. 2), the other one was overwhelmed by elastic and Compton scattering from the sample, and no indication of an edge was visible. Comparison of the near-edge spectrum with references of platinum metal, platinum oxide and platinum chloride measured during the same beamtime and with the same setup shows that the platinum in the soot sample is neither in its metallic state, nor an oxide, nor is it a mixture of the two (i.e., a linear combination of the metal and oxide spectra).

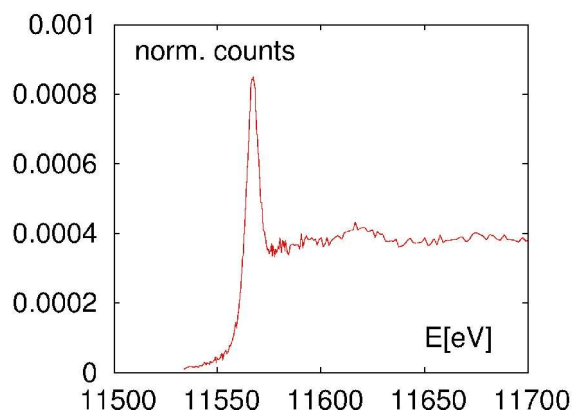


Fig. 2: Near-edge spectrum of Pt in soot, Pt L_{α1} fluorescence as selected by the analyzer over the incident energy. There were 10^{12} to 10^{13} atoms of Pt in the sample at a concentration of 100 ppm.

Discussion

The high-resolution fluorescence analyzer described here permits trace-element analysis at significantly lower concentration levels than previously possible with solid-state detectors. Apart from the background suppression demonstrated here, it is also capable of separating closely-spaced fluorescent lines, which is also highly important in trace-element analysis.

Acknowledgments

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References

- [1] K. Attenkofer, B.W. Adams, M.A. Beno, AIP Conf. Proc. 705, 1138-1141 (2004)
- [2] B.W. Adams, K. Attenkofer, to be submitted